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Influence of Bridgman solidification on microstructures and magnetic behaviors of a non-equiatomic FeCoNiAlSi high-entropy alloy

Tingting Zuo [a, *], Xiao Yang [a, b], Peter K. Liaw [c], Yong Zhang [a]

[a] State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China
[b] State Key Laboratory of Advanced Metallurgy, University of Science and Technology Beijing, Beijing 100083, China
[c] Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996, USA

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ABSTRACT

The non-equiatomic FeCoNiAlSi alloy is prepared by the Bridgman solidification (BS) technique at different withdrawal velocities (V = 30, 100, and 200 μm/s). Various characterization techniques have been used to study the microstructure and crystal orientation. The morphological evolutions accompanying the crystal growth of the alloy prepared at different withdrawal velocities are nearly the same, from equiaxed grains to columnar crystals. The transition of coercivity is closely related to the local microstructure, while the saturation magnetization changes little at different sites. The coercivity can be significantly reduced from the equiaxed grain area to the columnar crystal area when the applied magnetic field direction is parallel to the crystal growth direction, no matter what is the withdrawal velocity. In addition, the alloy possesses magnetic anisotropy when the applied magnetic field is in different directions.

1. Introduction

The microstructure and texture related to the processing of a metal were key factors that influence the final properties. An ever-increasing interest has been attracted to improve mechanical/physical properties by obtaining the desired microstructure and texture of materials using directional solidification [1–3]. During the directional solidification, due to the solute redistribution and texture rearrangement, solidification defects, such as lateral boundaries, are eliminated, which facilitates the formation of the columnar-crystal or single-crystal structure with the preferred crystallographic orientation, leading to the improvement of the longitudinal mechanical properties and magnetic properties of alloys [4]. Since both the grain orientation and grain size have effects on the magnetic properties of the soft magnetic materials, in which the basic requirements are high permeability, low coercivity, and small core loss. If the easy axis of magnetization is the same as the grain orientation, an external magnetic field parallel to the easy axis can increase the permeability drastically [5]. As to the grain size, if it is in the magnitude of micron, the large grain size is preferred due to the reduction of the coercivity [6,7]. Thus, for preparing the soft magnetic materials, the factors affecting the grain orientation and grain size should be considered.

In the past decade, high-entropy alloys (HEAs), which are defined as multi-component alloys containing several components (≥5) in equal or near equal atomic proportions [8], have attracted much research interest [9–11] because of their unusual properties, such as high hardness [12–14], high fracture toughness [15], high-temperature resistance [16], excellent tensile ductility [17,18], and super-plastic behavior [19]. Existing reports show that there is the potential for HEAs to be used in heat-resistant and wear-resistant coatings, diffusion barriers [20], mold linings, and areas that need excellent magnetic properties and high-temperature performance and so on. In our precious study [21], it is found that the as-cast $FeCoNiAl_{0.2}Si_{0.2}$ alloy shows excellent soft magnetic properties and good plasticity, although its coercivity is somewhat higher. The subsequent heat treatment can largely reduce the coercivity by decreasing the internal stress. Besides this process, other methods, which favor to form the texture or increase the grain size, can also improve the magnetic property. One technique is the directional solidification. In this study, the properties of the $FeCoNiAl_{0.2}Si_{0.2}$ alloy prepared by Bridgman-type-solidification (BS) are investigated. The relationship between the microstructures and magnetic properties is explored.

* Corresponding author.
E-mail address: zuott1986.520@163.com (T. Zuo).

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2. Materials and methods

In this paper, the alloy ingot of nominal compositions of $FeCoNiAl_{0.2}Si_{0.2}$ (0.2 is in a molar ratio) was synthesized by arc-melting pure elements with a purity higher than 99.95 weight percentage (wt. %) under a high-purity argon atmosphere on a water-cooled Cu hearth. The alloy was remelted at least four times to obtain the chemical homogeneity. Cylindrical rods with a diameter of 3 mm were prepared by suction casting. Then, the cylindrical rod was placed in an alumina tube with an internal diameter of 3 mm and inductively heated to the completely melting condition by adjusting the heating power, holding for 20 min. The BS was carried out with different withdrawal velocities (V) (V = 30 μm/s, 100 μm/s, and 200 μm/s, and the corresponding alloy samples are simply defined as A30, A100, and A200, respectively) through a temperature gradient (G) of about 45 K/mm into the water-cooled Ga-In-Sn liquid alloys. The BS equipment is the same as reference [22].

The cylindrical samples after BS were cut open along the longitudinal direction and, then, ground, polished, and etched. The microstructure evolution of the synthesized specimens was investigated by a metallographic microscope. Crystal structures were identified, using an X-ray diffractometer (XRD) under radiation conditions of 30 kV and 20 mA, with a Cu target and a scanning speed of 10°/min. The orientations of the grains were determined by the electron back-scatter diffraction (EBSD), using a CAMBRIDGE S-360 scanning electron microscope (SEM). Before the EBSD characterization, the HEA bulk sample was cross-sectioned using the electric discharge machining, and finally polished using the 1.0 μm diamond paste. In order to study the magnetic property, cylinders of 3 mm in diameter and 1 mm in thickness were prepared from the directional solidified rods and tested by an LDJ 9600 vibrating sample magnetometer (VSM) at room temperature. Then (1) the saturation magnetization, Ms, which is the largest magnetic moment that the alloy can possess when the applied field is increasing, (2) the remanence, Mr, which remains when the applied field is restored to zero, and (3) the coercivity, Hc, which is the reverse field needed to reduce the magnetization to zero, are marked on the hysteresis loop.

3. Results and discussion

Fig. 1 shows the XRD patterns of the samples prepared at different withdrawal velocities. It is clear that all directional solidified samples are with a single FCC structure which is not influenced by the withdrawal velocities. However, compared with the directionally solidified samples, the as-cast sample shows a minor peak besides the (111) face-centered-cubic (FCC) peak, which proved to be a body-centered-cubic (BCC) phase. It implies that the process of directional solidification can suppress the formation of BCC precipitates.

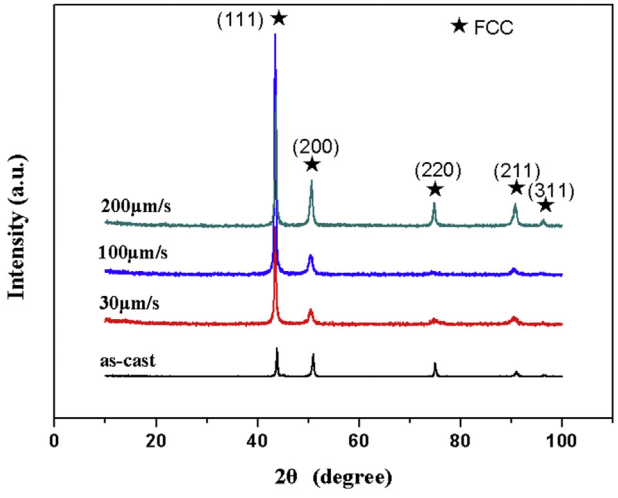

Fig. 1. The XRD patterns of the samples prepared by arc melting and by BS with different withdrawal velocities.

The optical microstructure evolution of the $FeCoNiAl_{0.2}Si_{0.2}$ alloy prepared by BS at the withdrawal velocity of 200 μm/s is presented in Fig. 2. It is clear that the microstructure is composed of three parts, which is defined as the initial region (the bottom area, which is the unstable area), the transition zone (the middle area), and the stable area (the top area). The crystal growth direction is shown in the lower right corner of Fig. 2. The microstructure transforms from the polycrystalline morphology at the bottom to dendrite morphology at the top, although there are some defects. Unlike the dendrites at the middle place growing to different directions, only two different grain orientations can be found at the top place, which implies that directional solidification promotes preferred grain orientation. As known, the microstructure of directional solidification mainly depends on the temperature gradient, G, and the process parameters, such as the growth velocity, V, in addition to the original solidification characteristics of the materials [3]. Since in this experiment, the temperature gradient is relatively constant, the main factor that needs to be considered is the growth rate, which is determined by the withdrawal velocity. For different withdrawal velocities, the microstructure transformation process is similar, which transfers from the polycrystalline to dendrite morphology. But the grain sizes for different withdrawal velocities are different, although there is no obvious relationship between the grain size and withdrawal speed.

The magnetic properties of the BS samples are plotted in Fig. 3. Similar to the as-cast alloy, the BS samples are ferromagnetic. But in different regions, the performances exhibit some differences. Saturation is usually regarded as being independent, to a large extent, of the microstructure. Small variations can, however, be detected for the A30 and A100 alloys in different parts of the samples. But the deviation for the A200 alloy is more obvious, since the fast withdrawal speed can lead to the instability of crystal growth. Overall, the saturation magnetization values are at the same level for different withdrawal velocities, which are in the range of 80 emu/g to 120 emu/g. Since the structure-insensitive saturation magnetization is largely dependent on the composition of the alloy, it is not changed substantially by different manufacturing process.

However, the structure—sensitive properties, such as the coercivity, permeability and hysteresis loss, are closely affected by the magnetization processes of domain nucleation, domain-wall motion, and domain rotation. These processes, in turn, are governed by the intrinsic properties, such as the saturation magnetization and magnetic anisotropy as well as microstructural details, including dislocations, grain boundaries, and precipitates [23]. It is well known that the grain boundary obstructs the movement of magnetic domain walls. Therefore, the specimens with the greater width of grains and lower densities of grain boundaries have the lower coercivity. The coercivity depends on the grain size, as shown below [6,24]:

$$H_c \approx 3\sqrt{\frac{\kappa_B T_c K_1}{\alpha M_s}}\frac{1}{D} \quad (1)$$

where H_c is the coercivity, D the grain size, M_s the magnetization saturation, K_1 the magneto-crystalline anisotropy, κ_B the Boltzmann constant, T_c the Curie temperature, and α the lattice constant.